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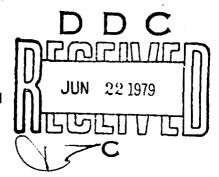


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Report 2273

EFFECTIVENESS OF STANDARD TEST FLUIDS
IN EVALUATING THE FUEL RESISTANCE OF ELASTOMERS

by
Paul E. Gatza
Paul Touchet
Floyd P. Powell



April 1979

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U.S. ARMY MOBILITY EQUIPMENT
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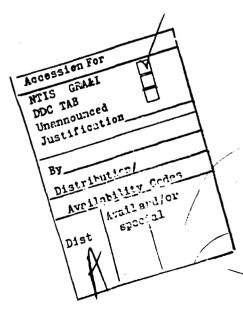
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EFFECTIVENESS OF STANDARD TEST FLUIDS IN

EVALUATING THE FUEL RESISTANCE OF ELASTOMERS

I. INTRODUCTION

- 1. Subject. This report details investigations conducted and results obtained in efforts to evaluate the reliability of standard reference or test fuels used to determine the resistance of elastomeric compounds to deterioration in physical properties resulting from contact with aromatic and other deleterious constitutents of commercial gasoline and related fuels.
- 2. Background. Deterioration of rubber end items such as gaskets, O-rings, hoses, and coated fabrics used in fuel storage tanks is essentially proportional to the aromatic content of the fuel to which the elastomer is periodically or continually exposed. The technology of producing gasolines and their ultimate composition is constantly changing. Ecological factors, such as pollution consciousness, and the uncertainties associated with immediate and future sources of supply (grade and composition of petroleum resources) have further complicated the task of keeping pace with the state-of-the-art. Fuels having an aromatic content as high as 60% are known to be in use to satisfy certain engine performance requirements.

The evolution in recent years of three distinct classes of gasolines – leaded, low-leaded, and unleaded – to satisfy tighter exhaust emission control standards has created additional confusion. Additives placed in, or removed from, fuel to reduce emission pollution have confounded correlation of these deterioration/aromatic content relationships. Since these additives are in low concentrations, their contribution, if any, to accelerate rubber deterioration is practically impossible to discern.

Fuel resistance of elastomeric compounds is generally determined by measuring the amount of swelling and deterioration of physical properties after immersion of specimens in standard reference fuels of known aromatic content. These reference fuels are detailed in ASTM Method D-471 and Federal Test Method Standard (FTMS) 601, Method 6001. ASTM Reference Fuel A of D-417 corresponds to Medium No. 4 in Method 6001 and is composed of 100% isooctane. Reference Fuel B of D-417 consists of 70% isooctane and 30% toluene, by volume, and is equivalent to Medium No. 6 in Method 6001. D-471 also lists a Reference Fuel C which is 50/50 isooctane/ toluene by volume. The nearest Method 6001 equivalent is Medium No. 5, a blend of isooctane, toluene, xylene, and benzene in a 60/20/15/5 ratio by volume. Method 6001 also references Mediums 7, 8, and 9 which consist of 100% benzene, toluene, and xylene, respectively.

Recent discoveries regarding the carcinogenic nature of benzene and subsequent OSHA directives forbidding its further use have disqualified Medium 5 of Method 6001. Thus, at present there is no accepted reference fuel containing 40% aromatic hydrocarbons.

The objectives of this program were, therefore:

- a. To evaluate the fuel resistance of representative elastomeric compounds exposed in commercial leaded, low-leaded, and unleaded gasolines.
- b. To ascertain whether relationships could be established between the severity of deterioration and the relative content of aromatic constituents or other additives in the gasolines.
- c. To determine the relevance of the various aromatic components of Medium No. 5 and to determine their individual contribution to elastomer deterioration.
 - d. To recommend a satisfactory alternative in lieu of Medium No. 5.
- e. To provide recommendations which would effect adoption of a standard series of fuels accepted by both ASTM and DOD.

II. INVESTIGATION

- 3. Scope, Work under this project was divided into two phases. The first phase encompassed the determination of the extent of deterioration in physical properties of clastomeric compounds exposed to commercial fuels and gasolines of known composition. These fuels included leaded, low-leaded, and unleaded types. The second phase concerned similar determinations, employing laboratory-prepared test fuels, both standard and nonstandard, of varying known aromatic content. Fuels and materials used in each phase are detailed as follows:
- a. Phase I 34 leaded, low-leaded, and unleaded fuels were used in this investigation. They were obtained by various means—from producers, other government agencies, and laboratories—and to maintain randomness, were purchased at service stations in the Washington, D.C. metropolitan area. Additionally, Reference Fuels B and C of ASTM D-471 and Medium No. 5 of FTMS, Method 6001, were included as typical currently used reference standards. These fuels, their sources, classes, and code identifications for further reference are shown in Table 1.



	Table 1. Phase I	Fuel Identification		
Fuel Nomenclature	Company	Refinery	Fuel Class	Code
1 Arco Clear	Atlantic-Richfield	Houston, 1X	Unleaded	AR
2. Arco Clear	Atlantic-Richfield	Philadelphia, PA	Unleaded	AR-
3. Arco Clear	Atlantic-Richfield	Wilmington, CA	Unleaded	AR
4. Sun's no-lead experimental gasoline, Sample No. 1	Sun Oil Co.	Philadelphia, PA	Unleaded	S-1
5. Sun's low-lead experimental gasoline, Sample No. 2	Sun Oil Co.	Philadelphia, PA	Low-Leaded	8-2
6. Shell-of-the Euture	Shell Oil Co.	Wood River, II	Unleaded	SH I
7. Amoco	American Oil	Purchased locally	Unleaded	AM-
8. Amoco Super Premium	American Oil	Purchased in Fredericksburg, VA	Unleaded	AM-
9. Amoco	American Oil	Purchased in Fredericksburg, VA	Unfeaded	AM
10 Amoco Super Premium	American Oil	Chicago, II	Unleaded	AM-
11. Amoco	American Oil	Chicago, H	Unloaded	AM-:
12 Amoco 940 regular grade to meet VVG-1690	American Oil	Chicago, II	Unleaded	AM (
13. American 945 regular grade to meet VVG-1690	American Oil	Chicago, H	Low-Leaded	AM

Falsla 1	Divariat	Fuel Identification (Cont'd)
13DE I	FD.IN' I	rue denimenton (Contab

Fuel Nomenclature	Company	Refinery	Fuel Class	Code
14 Mobile Pre- mum Sample 37143	Mobile Oil Corp.	Chicago, II	Leaded	M-i
15. Mobil Regular Sample 37144	Mobil Oil Corp.	Chicago, IL	Leaded	M-2
16. Mobil Special Sample 37145	Mobil Oil Corp.	Chicago, IL	Low-Leaded	M-3
17. Texaco Lead- Free Code 365LAP- 297	•	Los Angeles, CA	Unleaded	T-1
18. Texaco Fire Chief Code 370LAP-298	Texaco, Inc.	Los Angeles, CA	I ow-Leaded	T-2
19. Texaco Sample No. CCL-F-712	Texaco, Inc.	Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Low-Leaded	1-3
20. CRC Sample No. CCL-F-743		Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Unleaded	C-1
21. Howell Sample No. CCL-F-744		Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Low-Leaded	HO-1
22. Esso Big Plus	Humble Oil	Bayonne, NJ	Low-Leaded	H-1
23. JP-4		Obtained from Fuels Handling Div of MFRADCOM	Unleaded	J-1

Fuel Nomenclature Company	Refinery	Fuel Class	Code
24. Mogas	Obtained from Fuels Handling Div of MERADCOM	Leaded	G-1
25. Reference Fuel – B of ASTM D-471	Prepared in Laboratory		В
26. Reference Fuel C of ASTM D-471	Prepared in Laboratory		FC
27. Med. No. 5	Prepared in Laboratory		11
28. Sample No. CCL-F-755	Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Leaded	C-5
29. Sample No CCL-F-766	Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Leaded	C-6
30. Sample No. CCL-F-777	Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Unleaded	C-7
31. Sample No. CCL-F-785	Obtained from Coating & Chemical Lab, Aberdeen Prov- ing Ground, MD	Unleaded	C-8
32. Amoco, — American Oil AFURI Code No. 3894	Obtained by South- west Research from Texas City, TX	Unleaded	SW-1
33. Union, Union Od AFLRF Code No. 4109	Obtained by South- west Research from San Francisco, CA	Low-Leaded	SW-2

Fuel Nomenclature	Company	Refinery	Fuel Class	Code
34. Texaco, AFLRF Code No. 4138	Texaco	Obtained by South- west Research from Los Angeles, CA	Low-Leaded	SW-3
35. Chevron, AFLRF Code No. 4013	Chevron	Obtained by Southwest Research from Oakland, CA	Low-Leaded	SW-4
36. Blend I, Amoco Indolene Clear, AFLRL Code No. 4145	Amoco	Obtained by Southwest Research. Described as 100% Indolene Clear	Unleaded	SW-5
37. Blend 2		Blended by South- west Research con- taining 86% Indolene clear (Blend 1) and 14% aromatic blend (composed of 44.3% toluene, 17.5% xylenes, and 38.2%	Unleaded	SW-6

aromatic solvent No. 100 - boiling range

Blended by South-

west containing 70%. Indolene clear and 30% aromatic blend (see item 37 for blend composition).

Unleaded

SW-7

316-350°F).

38. Blend 3

Gas chromatography and other laboratory analytical procedures were employed to obtain a breakdown of each fuel in terms of aromatic concentration by type or chain structure—and tetrethyl/tetramethyl lead content. This information is contained in Table 2. A good representative range of aromatic content was obtained—from a low 8.1% to a relatively high concentration of 62.9%.

Seven elastomeric compounds, representative of those used in applications requiring fuel resistance, were selected, mixed, and vulcanized as 6- by 6-inch test sheets having a thickness of about 0.080 inch. Formulations and curing conditions for these compounds are shown in Table 3.

Fuel-resistant elastomers also play a significant role in military applications as coated fabrics in fuel storage tanks and related end items. Therefore, two fabric-coating materials, a polyether urethane and a polyester urethane, were also included in this study. These materials, whose formulations are proprietary, were supplied in large cured sheets by Uniroyal, Inc., the manufacturer.

- b. Phase II is two compounds, the polysulfide and the medium NBR (designated J-232 and J-234), were not included in this phase of the program. Otherwise, formulations and sample preparation were the same as in Phase I. Sufficient materials were prepared initially in the laboratory or were obtained from Uniroyal, Inc. to conduct all tests under both phases. Fuels used in Phase II were the standard ASTM D-471 or FTMS 601, Method 6001 reference fuels or variations thereof. The variations were selected to ascertain more precisely the relationships between toluene, xylene, and benzene as are encountered in fuels such as Medium No. 5. Additional data obtained could then be subjected to analysis by computerized reduction techniques. Composition of the Phase II fuels is shown in Table 4.
- 4. Tests Conducted. Initial physical properties—tensile strength, clongation, 200% modulus, and Shore A hardness—were determined according to procedures detailed in ASTM D-412 and ASTM D-2240. Volume swell after 4-, and 7-day room temperature immersion for Phase I and Phase II tests, respectively, was determined according to Method 6211 of FTMS. Retention of tensile strength and clongation after immersion was ascertained according to FTMS 601, Method 6111, with values obtained based on the swollen cross sectional area per paragraph 4,8,1 of Method 6111. The diffusion rate tests (Phase I only) were performed in accordance with the provisions of MII-F-52573, paragraph 4,6,2,2, typically used to evaluate coated-fabric materials for fuel tanks. Test procedures followed were similar for both phases of the program. However, all fuel immersion tests were not conducted in either phase. Data obtained were deemed sufficient to substantiate conclusions and recommendations set forth later in this report.

Fuel Code No	Aromati By GC	ie –		Ethyl e Benzen (%)	M +	P, ne X	O ylene	TI		C,	
C-7	62.9	.8	5.1	5.4	14.		(%) 5.8	_		(%) 23,9	
AM-2	56,5	.2	29.0	2.2	7.1		2.3			9,7	_
AM-I	55,3	.4	33.2	1.6	5.2	: :	2.2	.o	7	8.1	4.4
SW-7	52.1	.7	22.6	2.1	4.6	. 2	2.4	>.0	ì	12.9	5.0
FC	50.0		50.0	*Name	farma		-	-			- 1.5
C-1	49.8	.4	23.9	2.1	7.4	2.	.4	.02	25	7.7	5.8
AM-4	48.4	.1	23.4	.7	2.0	1.	1			2.5	8.5
SW-1	46.4	.8	15.9	2.9	10.8	3.	4	>.01		2.44	0. 3
10-1	45,7	3.6	16.5	2.1	9,5	2.9		.24		6.5	4.3
W-6 ·	41.9	.7	20.5	1.4	3.8	1.8	} :	÷01	•	3,7a	7.3
-3	41.4	1.6	9.9	2.6	10.9	3.6		.04		 7.8	4.9
W-2	40.6	.9	6.9	1.9	8.2	3.0			19		
4	40.0	5.0	20.0	• ***	15.0						• .
1 3	18.9	1.5	9.8	2.6	10.2	3.5		.02	9,	i	1.8
2 3	8.6	.9	5.6	2.1	8.0	2.1		40	9.).6
3.	8.6	1.5	7.0	2.0	8.3	2.8			7.		,9
-3 30	5.4	.7 2	4.7	1.5		2.4		35 ;			
-3 30	5.4	.5 5	i.6 j	2.3		3.4)45			•
3 35	5.2	1.2 6	.8 1			2.6	.0		9.4 8.8		

Table ?	Composition	of Liquid	Hydromehon	Fueld (Cont'd)
Table 2.	Composition	oi Liauia	rivurocarbon	rucia (Conta)

	Total			quiu ityu					
	Aromatic	_		-	M → P		TEL/		_
Fuel	By GC			Benzene	=	-			C ₁₀ +
Code No.		(%)			(%)		(%)		(%)
H-1	33.5	1.7	7.4	1.7	6.2	2.3	.44	7.2	6.9
AR-I	33.1	1.1	12.4	1.1	3.2	1.1	.01	7.3	6.9
C-8	32.9	.4	18.6	.7	3.3	1.2	. -	3.1	5. 6.
AM-6	32.6	1.0	11.9	1.4	3.9	1.6	.005	8.0	4.6
M-3	31.1	1.0	4.5	2.7	7.1	2.6	.20	7.7	5.3
SH-1	30.8	1.4	10.8	2.0	4.9	2.0	.02	5.6	3.9
M-1	30.7	1.6	5.6	2.3	5.6	2.1	2.50	7.9	5.4
SW-5	30.0	.9	12.9	1.0	2.9	1.2	.01	11.12	·
В	30.0	-	30.0	_	_		-		-
S-1	28.9	1.1	5.6	1.2	4.7	1.8	.015	6.0	8.4
S-2	27.9	.9	3.9	.9	3.9	1.4	.41	6.7	10.1
SW-4	27.0	1.2	5.5	2.8	4.0	1.6	.44	12.0°	
G-1	26.7	.8	4.0	1.3	4.6	1.7	.84	6.3	7.9
AM-7	25.1	1.5	6.2	1.8	5.1	2.0	.40	5.9	2.5
AM-5	24.4	1.5	6.0	1.7	4.9	1,9	.007	5 5.7	2.6
J-1	13.5	.3	.7	.3	1.1	.4	.025	1.7	9.1
C-6	12.4	.3	1.7	.6	3.3	1.0	2.26	3.7	1.7
C-5	8.1	.5	2.2	.4	1.5	.5	2.68	1.4	1.6

NOTES:

a C,+

Table 3. Test Compound Formulations

			Part	s by We	ight		
Ingredients	J-229	J-230	J-231	J-232	J-234	J-235	J-236
Hydrin 200 (ECO Rubber)	100.	*					~
Thiokol ST (Polysulfide)				100.	,		
Neoprene WRT Rubber		100.					
Paracril AJ (Low NBR)	,		100.				
FR-N 501 (Med NBR)			· ·	٠.	100.		
Hycar 1001 (High NBR)	¥	,	•		•	100.	
Paracril OZO (NBR/PVC)							100.
Magnesium Oxide		4.0	***	- -		-	er ente
Stearic Acid		0.5	1.0	1.	1.0	. 1.0	1.0
Zinc Oxide	~	5.0	5.0	**	5.0	3.0	5.0
NBC (Nickel dibutyldithio- carbamate)	1.	-		- mily	·		
Zinc Stearate	. 1.	Autor	1944				
Neozone A (N-phenyl-alpha- naphthylamine)	****	2.0	-		-		-
HAF, carbon black		50.		***	•		us ya
SRF, carbon black		.ma	50.	60.	50.	50.	50.
FEF, carbon black	30.			··	-	**	
Lime	-	***		1.	· .		*140m
Red Lead	5.		W 4 Pr				
FP-95, Plasticizer	, with	÷		10.			

Table 3. Test Compound Formulations (Cont'd)

Table 3. T	est Com	pound	Formula	tions (C	ont'd)		
			Par	ts by W	eight		
Ingredients	J-229	J-230	J-231	J-232	J-234	J-235	J-236
Sulfur	••		1.5		1.5	1.5	1.5
Zinc Peroxide				5.			
MBTS (2.2' Benzo Thiazyl disulfide)	-		1.5		1.5	1.5	1.5
NA-22 (2-mercaptiomidazoline	1.5	.5	, ,				<u>-</u> .'
Antioxidant MB (2-mercapto benzimidazole)	1.5						
Curing Conditions							
Temperature, "F	320	310	310	300	310	310	310
Time, minutes	30	30	30	40	30	30	30

					able 4.	Table 4. Phase II Test Fuels	Test	uels					
						Perce	int by	Percent by Volume					
III SHOOTH	-	C I	m	4	6	4	-						
Sooctane 100	100					,	-	×	6	10	=	12 13	=
				ŀ	Ö,	60. 60. 60. 60. 60. 60.	.09	.09	.09	60	99	50	4
oluene	i"	100.		1	30.	יי 30. יי 30. יי	۶	7	4,	•		j	?
				:		;	į	<u>.</u>	Ċ.	40.	5. 40. 25. 30. 40	30.	40
, ICH	1	;	<u>.</u>	ł	,	15.	5.	5. 20. 15.	15.	!	31 31 31	. 5	-
lzene			i	100.	· !	5.	15.	5. 15. 5. 20	20	ı	•	<u>:</u>	<u>.</u>

5. Results. Original physical properties for all compounds are shown in Table 5. Tables 6 through 14 contain tensile strength retention, elongation retention, volume swell, and diffusion data for all Phase 1 tests. Table 15 gives volume swell data for each of the seven elastomer types after immersion for 7 days in one of the fourteen Phase II media. Tables 16 and 17 contain percent tensile strength and elongation retention values after similar immersion of dumbbell specimens in six of the fuels. Table 18 compares actual and predicted volume swell data for tested and untested reference fuel/rubber combinations.

III. DISCUSSION

6. Phase 1. The data for total aromatic content of the 34 different fuels (Table 2) underscore the wide variation in composition and ultimate deleterious effects on elastomeric compounds observed in this investigation. When categorized according to lead content, the unleaded and low-leaded fuels display the widest range -24.4% to 56.5% and 8.1% to 62.9%, respectively. The leaded fuels occupy a narrower mid-range area -26.7% to 38.6% aromatic content. As aromatic content decreases, a proportionately greater amount of these components are classified under C_9 and C_{10} groupings, the exact structure of which is indeterminate.

The data in Tables 6 through 14 were organized as input to in-house computerized regression analysis software routines. It was hoped that this analysis would uncover certain patterns or relationships linking a particular aromatic constituent with the severity of deterioration observed for one, several, or all elastomers. No satisfactory correlation could be established. More sophisticated techniques, involving stepwise linear regression, were then applied. Here again, results were inconclusive. Aromatic groups (variables) removed early from the step wise analysis of one rubber, were retained until the end for another rubber. Also, in most cases, regression was complete after few passes.

Data for those rubber/fuel exposures which were employed throughout the Phase I study were plotted as bar graphs for each of the nine rubbers. Percent tensile retention and volume swell for these unleaded, low-leaded, leaded (where included), and test fuel/rubber combinations are shown in Figures I through 9. While no distinct pattern applicable to all rubber fuel combinations is discernible, certain generalizations can be made. Unleaded fuels tend to produce greater tensile strength loss and higher volume swell than low-leaded or leaded fuels. In some cases (all NBR's, neoprene, and the two urethanes), tensile loss is somewhat greater than that observed for the three test fuels, which also contained no lead. In the four situations where low-leaded and fully leaded fuels can be compared, results are mixed, with the presence of additional lead having no correspondingly greater adverse effect on tensile retention or volume swell.

**

Table 5. Original Physical Properties of Test Compounds

Compound	Tensile Strength (lb_in ²)	Flongation (%)	200% Modulus (!!s/m²)	Hardness Shore A
J-229-FCO	1850	360	1060	65
J-230-Neoprene	3298	235	2830	75
J-231-Low NBR	2030	180		7.5
J-232-Polysulfide	741	200		71
J-234-Med/NBR	1880	345	800	68
J-235-High NBR	2640	425	1270	70
J 236-NBR/PVC	2270	220	2120	85
Ester Urethane	3160	555	840	85
Ether Urethane	4050	460	1410	85

Table 6. Properties of Epichlorohydrin-Ethylene Oxide (ECO-1) Rubber Exposed to Liquid Hydrocarbon Fuels

Fuel Code No.	Fotal Aromatic By GC (+)	Type of Gasoline	Tensile Retained ('.)	Flongation Retained (17).	Volume Swell (T)	Diffusion Rate (oz/yd²/day)
AM-2	56.5	UL.	49,0	52.8	40.1	.6909
SW-7	52.1	UL	50.4	55.6	25.4	.464.3
FC	50.0	T	45.8	47.2	39,6	.6840
SW-1	46.4	ÜL	56.7	61.1	25.1	.4111
SW-6	41.9	Į!Į	54.4	62.5	25.4	2860
SW-2	40.6	11.	61.6	69.4	23.5	.2341
11	40.0	T	56.5	52.3	27.4	.3439
SW-3	36.4	11	63.1	66.7	22.3	.1757
SW-5	30.0	u	63.9	70.8	19.1	.1747
В	30.0	T	62.8	63.9	21.5	.2390
SW-4	27.0	1.1	71.4	75.0	18.0	.1156

Fuel Code No.	Total Aromatic By GC (27)	Type of Gasoline	Fensile Retained (%)	Flongation Retained (%)	Volume Swell (7)	Diffusion Rate (oz/yd²/day)
AM-2	56.5	UL	23.9	42.9	97,9	2.6512
SW-7	52.1	UL	25.4	47.0	98,9	2.1623
FC	50.0	T	25.0	38.0	9.30	1.5670
SW-1	46,4	vi.	23.9	47.0	95.0	2.6814
SW-6	41.9	UL	22,6	42.9	85.0	1,994,3
SW-2	40.6	LL	23.0	42.9	87.9	2.6697
H	40.0	r	27.0	47.0	74.9	1.2210
SW-3	36.4	n.	26.6	47.0	82.7	2.4486
SW-5	30.0	uL	30,8	57.0	66.7	1.3189
В	30.0	T	33.1	57.0	61.5	0.9020
SW-4	27.0	1.1.	32.1	52.4	61.3	1.6388

Table 8. Properties of Low Acrylonitrile Rubber Exposed to Liquid Hydrocarbon Fuels

		hxposed to	Liquid Hy	drocarbon Fi	iels	
Fuel	Fotal Aromatic By GC	Type of	Tensile Retained	Flongation Retained	Volume Swell	Diffusion Rate
Code No.	(°.)	Gasoline	(°a)	(5)	(6)	(oz:yd²/day)
(°-7	62.9	LL	22.9	35.9	102,9	2.320
AM-2	56.5	UL	18.2	32.8	90.5	2.621
AM-1	55.3	UL	21.3	39.1	95,5	2.624
SW-7	50.8	UL	18.6	34.4	85.3	2.124
FC	50.0		27.2	45.3	84.1	1.535
C-1	49.8	UL	16.2	32.8	78.9	1.544
AM-4	48.4	UL	19.3	39.1	88.9	1.855
SW-1	46,4	ÜL	20.5	34,4	80.5	2.237
HO-1	45.7	LL	21.7	40.6	79.4	1.709
SW-6	41.9	UL	27.4	48.4	70.3	1.621
T-3	41.4	LL	27.4	46.9	72.2	2.040
SW-2	40.6	LL	24.2	42.2	73.3	2.125
П	40.0		27.5	46.9	65.4	1.133
T-1	38.9	UL	20.4	40.6	81.1	2.186
T-2	38.6	L	23.7	45.3	75.6	1.788
AR-2	38.6	UL	28.7	48.4	60.2	1.364
SW-3	36.4	LĹ	25.0	45.3	65.9	1.710
AM-3	36.4	UL	27.0	45.3	64.1	2.055
AR-3	35.2	UL	23.4	43.7	66.0	1,405
H-1	33.5	LL	24.2	46.9	73.6	1.720
AR-1	33.1	UL	27.0	50.0	60,5	1.182
C-8	32.9	UL	27,9	51.6	64.2	1.611
AM-6	32.6	UL	23.9	43.8	68.5	1.389
M-3	31.3	LL	23.2	45.3	60.5	0.907
SH-1	30.8	UL	23.7	45.3	62.2	1.407
M-1	30.7	L	30.2	53.1	60.5	0.779
SW-5	30.0	UL	29.0	48.4	56.3	1.120
В	30.0		28.6	48.4	54.3	0.903
S-1:	28.9	UL	25.1	45.3	60.3	0.927
S-2	27.9	LL	29.0	48.4	58.4	s 0.961
SW-4	27.0	LL	27.1	43.8	54.3	1,215
G-1	26.7	L	34.0	53.1	53.1	1.151
AM-7	25.1	LL	26.5	45.3	57.5	1,141
AM-5	24.4	UL.	30.3	50.0	59.5	1.118
J-1	13.5		58.3	73.4	30.8	0.150
C-6	12.4	LL	45.4	64.1	38.1	0.558
C-5	8.1	L.L	31.3	54.7	36,4	0.695

Table 9. Properties of Polysulfide Rubber Exposed to Liquid Hydrocarbon Fuels

Fuel Code No.	Total Aromatic By GC (%)	Type of Gasoline	Tensile Retained (%)	Elongation Retained (%)	Volume Swell (7)	Diffusion Rate (oz/yd²/day)
AM-2	56.5	UL.	64.9	68.0	10.7	.2271
SW-7	52.1	UŁ	69.6	72.0	8.7	.1837
FC	50.0	Ŧ	52.2	56.0	10.9	.2114
SW-1	46.4	UL	72.5	72.0	8.7	.1493
SW-6	41.9	UL	75.8	80.0	6.7	.1305
SW-2	40.6	LL	68.2	80.0	5,9	.1120
11	40.0	T	63.0	64.0	6.4	.0979
SW-3	36.4	LL	80.6	88.0	5.2	.0745
SW-5	30.0	UL	77.1	80.0	4.0	.0937
В	30.0	Ť	70.2	76.0	4.4	.0831
SW-4	27.0	LL	82.6	72.0	3.8	.0703

Table 10. Properties of Medium Acrylonitrile Rubber Exposed to Liquid Hydrocarbon Fuels

Fuel Code No.	Total Aromatic By GC ('7)	Type of Gasoline	Tensile Retained	Elongation Retained (%)	Volume Swell	Diffusion Rate (oz/yd²/day)
AM-2	56.5	UI	23.6	42.7	82.1	1.7162
SW-7	52.1	UL	26.6	53.3	66.4	1.0720
FC	50.0	T	28.2	48.0	65.6	1.2747
SW-1	46.4	UL	24.4	46.7	60.8	1.2180
SW-6	41.9	Ul.	28.3	54.7	51.9	.8060
SW-2	40.6	l.L	24,9	52.0	53.5	.8870
11	40.0	T	31.9	56.0	50.5	.8831
SW-3	36.4	1 L	36.6	62.7	147.2	.7175
SW-5	30.0	tit.	36.8	62.7	42.2	.5870
В	30.0	t	32.8	53.3	41.8	.5449
SW-4	27.0	11.	33.6	57.3	39,3	.5000

Table 11. Properties of High Acrylonitrile Rubber Exposed to Liquid Hydrocarbon Fuels

	Total	i.sposta to	Elquid Hy	arocarbon ru	icis	
	Aromatic	Type	Tensile	Flongation	Volume	Diffusion
Fuel	By GC	of	Retained	-	Swell	Rate
Code No.	('()	Gasoline	(6)	(6)	(6)	(oz/yd²/day)
C-7	62.9	LL	33.8	60.7	41.3	0.317
AM-2	56.5	ÜL	25.7	55.4	36.2	0.511
AM-I	55.3	UL	25.5	51.8	42.0	0.508
SW-7	50.8	ÜL	28.0	53.6	37,8	0.264
FC	50.0		29.3	51.8	40.2	0.535
C-1	49.8	UL	39.0	69.6	34.2	0.232
AM-4	48.4	UL.	29.3	62.5	35.4	0.298
SW-1	46.4	UL	30.7	55.7	34.8	0.220
HO-1	45.7	LL	30.5	62.5	33.2	0.234
SW-6	41.9	UL.	41.5	69.6	29.8	0.165
T-3	41.4	11.	53.2	78.6	30.8	0.144
SW-2	40.6	L.L.	36.4	58.9	28.7	0.000
11	40.0		36.9	64.3	30.4	0.191
Г-1	38.9	UL	38.9	66.1	30.5	0.148
T-2	38.6	i.	44.6	73.2	25.4	0.053
AR-2	38.6	UL.	45.7	73.2	27.5	0,077
SW-3	36.4	LL.	47.5	75.0	25.4	0.052
AM-3	36.4	UL	51.1	78.6	24.7	0.032
AR-3	35.2	UL	56.4	83.9	26.0	0.070
11-1	33.5	LL	58.3	83.0	27.8	0.097
AR-1	33.1	Ul	40,0	75.0	27,4	0.074
C-8	32.9	UL	43.0	71.4	25.7	0.118
AM-6	32.6	UL.	41.7	67.9	25.2	0,000
M-3	31.1	LL	60.1	87.5	21.7	0.015
SH-1	30.8	UI	48.3	76.8	23.2	0.042
M-1	30.7	l	48.3	73.2	20.8	0.004
SW-5	30.0	UL	54.0	80.4	21.9	0.054
В	30.0		45.4	73.2	25.4	0.116
S-1	28.9	UL	48.5	83.9	21.3	0.005
S-2	27.0	11	54.1	80.4	20.1	0.002
, SW-4	27.0	11	55,9	80.4	20.6	0.108
(i-1	26.7	i	52.1	87.5	15.8	0.005
4M- 7	25.1	11	52.8	82.1	19.5	0.010
AM 5	24.4	U	43.0	ϕ_{O} ϕ	18.4	0.026
J. l	13.5		878	101.8	4.1	0.003
C-6	12.4	11	80.1	98.2	6.6	0.008
C-5	8.1	11	- 14,8	. 0 <u>3</u> ,0	70	0.006

Table 12. Properties of Nitrile/PVC Blend Compound Exposed to Liquid Hydrocarbon Fuels

Fuel Code No.	Total Aromatic By GC (%)	Type of Gasoline	Tensile Retained (%)	Elongation Retained (%)	Volume Swell (%)	Diffusion Rate (oz/yd²/day)
AM-2	56.5	UL	28.0	42.4	65.9	1.3273
SW-7	52.1	UL	28.2	48.5	54.0	.9848
FC	50.0	τ	26.0	57.6	56.1	.926
SW-1	46.4	UL	41.8	72.7	50.0	.9440
SW-6	41.9	UL	50.9	84.8	42.8	.6488
SW-2	40.6	LL	40.5	75.8	43.2	.6183
11	40.0	T	46.0	60.6	42.0	,5554
SW-3	36.4	LL	50.3	75.8	39,9	.4697
SW-5	30.0	UL	56.3	109.0	35.2	.3380
В	30.0	T	62.7	90.9	37.3	.3776
SW-4	27.0	LL	45.7	63.6	33.0	.3170

Table 13. Properties of Polyester Polyurethane Coating Exposed to Liquid Hydrocarbon Fuels

Fuel	Total Aromatic By GC	Type of	Tensile Retained	Elongation Retained	Volume Swell	Diffusion Rate
Code No.	(9)	Gasoline	(%)	(%)	(%)	(oz/yd²/day)
C-7	62.9	LL	70.	100.9	9.9	.0039
AM-2	56.5	UL	59.5	105.0	13.3	.0800.
AM-1	55.3	UL.	53.8	8,001	16.4	.0140
SW-7	50.8	UL	62.9	100.9	11.7	.0120
F¢	50.0		58.5	98.3	16.9	.0400
C-1	49.8	UL	58.4	103.4	9.7	.0041
AM-4	48.4	UL	70.4	107.7	11.3	.0165
SW-1	46,4	UL	71.8	101.7	9.7	.0090
.HØ-1	45.7	LL	61.0	104.3	11.1	.0036
SW-6	41.9	UL	71.8	102.6	8.3	.0080
Т-3	41.4	LL.	69.2	104.0	8.3	.0029
S₩-2	40.6	LL	70.6	99.1	6.7	.0065
п	40.0		75.6	100.0	12.3	.0130
T-i	38.9	UL	67.3	102.5	7.7	.0050
T-2	38.6	L	73.1	105.1	5.8	.0023
AR-2	38.6	Ul.	66.6	107.7	6.8	.0070
SW-3	36.4	LL	78.3	100.9	5.3	.0070
ΛМ-3	36.4	UL	67.0	103.0	6.0	.0100
AR-3	35.2	UL	67.6	107.7	6.6	.0090
11-1	33.5	LL	66.0	105.0	10.4	.0030
AR-1	33.1	UL	66.3	99.2	7.6	.0100
C-8	32.9	UL	73.0	99.7	6.6	.0027
АМ-6	32.6	UL	81.1	101.7	7.2	.0006
M-3	31.1	LL	70.5	102.6	5.0	.0023
SH-I	30.8	UL	61.2	100.8	6.3	.0027
M-1	30.7	L ·	73.8	103.4	7.1	.0021
SW-5	30.0	UL	80.1	104.3	5.6	.0070
В	30.0	**	83.7	102.0	- 8.0	.0070 -
S-1	28.9	UL	71.4	104.3	6.8	.0060
S-2	27.9	LL	76.2	102.6	6.3	.0021
SW-4	27.0	LL	81.6	107.7	5.1	.0110
G-1	26.7	L	68.4	102.5	5.9	.0027
AM-7	25.1	L .	78.0	100.9	6,6	.0017
AM-5	24.4	UL	84.6	104.3	5.7	.0007
J-1	13.5	,	77.3	104.3/	2.1	.0020
C-6	12.4	LL	92.0	100.9	2.4	7,0002
C-5	8.1	LI.	8 <u>8.4</u>	100.9	3.4	.0026

Table 14. Properties of Polyether PolyurethaneCoating Exposed to Liquid Hydrocarbon Fuels

		Exposed to	Liquid H	drocarbon Fi	iels	
	Total	TD.	rge . s	\$18		** * ***
	Aromatic	Type	Tensile	Elongation	Volume	Diffusion
Fuel	By GC	of	Retained	Retained	Swell	Rate
Code No.	(%)	Gasoline	(%)	(%)	(%)	(oz/yd²/day)
C-7	62.9	"LL	35.8	79.2	62.2	1,674
AM-2	56.5	UL	41.3	85.2	60.2	1.844
AM-1	55.3	UL	42.1	87.8	62.6	1.510
SW-7	50.8	UL	43.2	84.3	57.8	1.660
FC	50.0		49.2	76.5	58,9	1.439
C-1	49.8	UL	46.6	93.0	54.5	1.644
AM-4	48.4	UL	42.2	86.6	52.8	1.325
SW-I	46,4	UL	44.4	80.9	54.3	1.718
HO-1	45.7	LL	46.6	91.3	54.5	1.539
SW-6	41.9	UL	47.2	86.1	49.6	1.298
T-3	41.4	LL	47.0	87.8	50.4	1.258
SW-2	40.6	LL	45.0	83.5	50.2	1.277
ii .	40.0	99.4	50.3	71.3	48.7	1.004
T-1	38.9	UL	49.4	91.3	51.7	1.261
T-2	38.6	L	50.6	93.0	48.0	1.081
AR-2	38.6	UL	46.8	86.1	49.1	1.180
SW-3	36.4	LL	46.3	82.6	47.3	1.196
AM-3	36.4	UL	50.5	90.4	47.5	1.036
AR-3	35.2	UL	49.2	89.6	48.3	1.129
H-1	33.5	LL	47.9	86.9	51.0	1.378
AR-1	33.1	UL	46.8	90.4	49.7	1.010
C-8	32.9	UL	45.4	91.5	46.2	1.162
AM-6	32.6	UL	48.8	93,4	43.0	1.092
M-3	31.1	LL	53.2	95.7	42.0	.855
SH-1	30.8	UL	51.8	94.8	43.7	1.100
M-1	30.7	L	53.1	87.8	45.6	.839
SW-5	30.0	UL	47.7	84.3	42.8	.979
В	30.0		52.7	71.3	42.3	.778
S-1	28.9	UL	54.4	90.4	44,6	.818.
S-2	27.9	LL	52.2	87.8	44.8	.878
SW-4	27.0	LL	51.9	87.0	41.1	.981
G-1	26.7	L	51.8	93.0	39.6	691
AM-7	25.1	LL	48.9	94.5	39.5	.936
AM-5	24.4	UL.	46.2	90,8	38.5	.813
J-1	13.5		58.7	97.4	24.8	.271
C-6	12.4	LL	55.4	98.4	30.8	.425
C-5	8.1	LL.	55.4	96.3	30.5	.664

Table 15. Volume Swell Phase II Tests

				Compoun	ds		
Fuei Code	FCO	Neoprene	Low NBR	High NBR	NBR PVC	Ester Urethane	Fther Urethane
. 1	.3	19	14	I	4	l	12
2	154	162	179	112	142	41	102
.3	112	. 167	164	90	123	20	91
4	187	157	181	131	157	50	114
5	22	62	54	25	37	8	. 42
6	28	78	67	32	43	16	47
7	31	81	70				
8	26	77	65				***
9	31	80	70				
10	30	78	68	- 31	44	18	46
11	20	77	64	30	43	15	47
12.	38	96	84	39	55	20	56
13	51	117	102	48	69	25	65
14	68	131	118	59	86	27	76

Table 16. Percent Tensile Strength Retention Phase II Tests

				Compoun	ds		
Fuel Code	FCO	Neoprene	Low NBR	High NBR	NBR/PVC	Ester Urethane	Ether Urethane
1	89	55	. 82	84	90	115	83
5	63	33	36	45	63	84	53
6	53	21	32	39	4.3	65	41
12	45	17	23	33	40	59	36
13	36	15	17	27	30	50	35
14	29	15	16	22	29	43	32

 Table 17. Percent Flongation Retention
 Phase II Tests

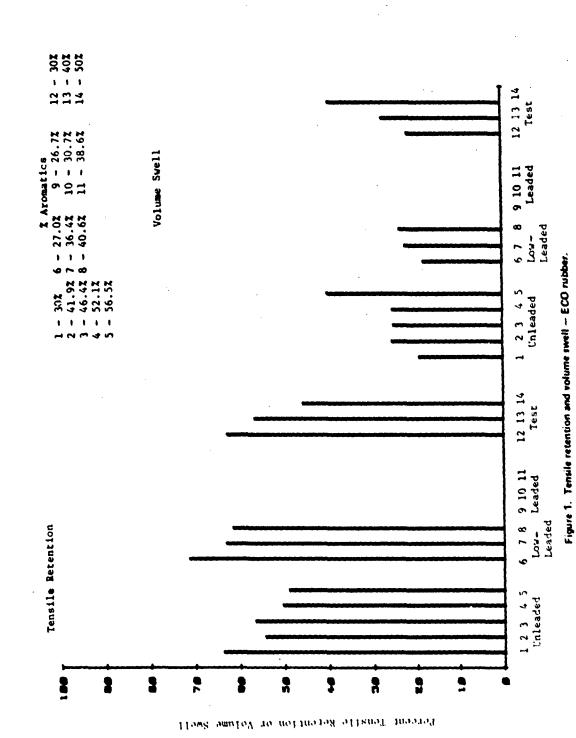
				Compoun	ds		
Fuel Code	FCO	Neoprene	Low NBR	High NBR	NBR/PVC	Ester Urethane	Ether Urethane
ı	88	77	89	77	110	98	101
5	64	57	58	7.3	91	102	71
6	6,3	45	58	57	89	108	91
12	56	40	50	52	82	105	89
1.3	40	38	36	45	64	102	9()
14	42	36	39	39	68	96	88

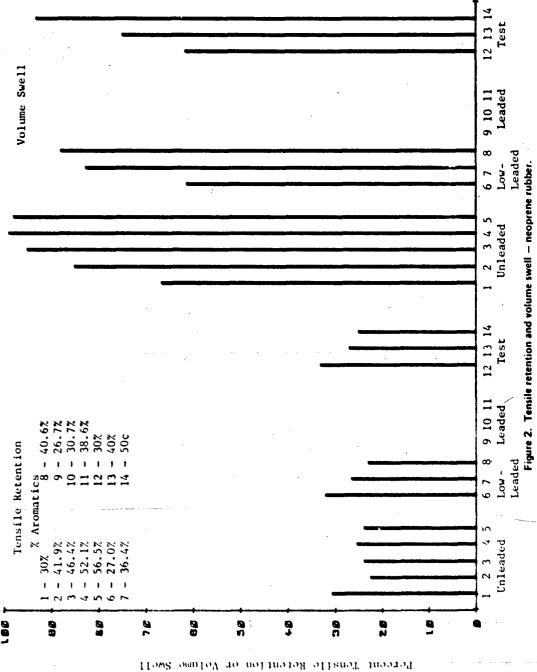
Table 18. Volume Swell Data - Actual and Predicted Values

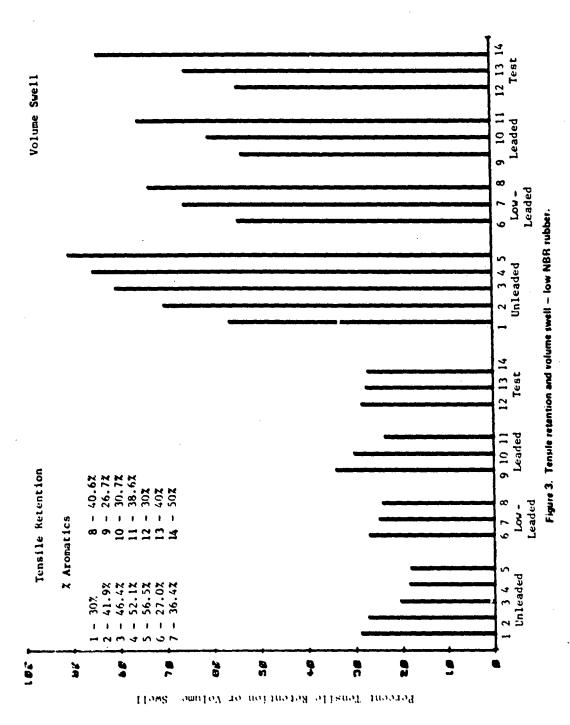
	Test Fuel		Composition Volume (2)	11110 (C2)			Actual and Fredicted Values	calcied val	nes		
Fuel								Volume Swell (%)	:11 (%)		
Code	Isooctane	Toluene Xylene	Xy!ene	Benzene	ECO	Neonrene	oan ao	Hist. Minn		Ester	Ether
	100	0	-	6	^	3113	TOM INDI		NBK/PVC	Urethane	Urethane
C	c	2	•	•	٠ :	<u>~</u>	4	-	4		1.2
۰~	•	3 °	>	-	154	162	179	12	142	17	: 2
, -	> (-	3	0	==	167	164	06	122		5 6
+	0	0	0	00 1	187	157	181	12.6		6 7	16
1 7,	70	30	0	C	,	(4	5	151	12/	29	_ 4
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	9) (> <	9	†	89	09	=	36	9.	لا
X 6	9	? ?	> 9	o (30	78	89	31	44	<u>×</u>	46
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	09	20	; =	ر د	, ;	ξ 5	ç	30	4	<u>&</u>	49
~	99	2 6	2	י נ	2	× 1	/3	36	42	13	4
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: '	2 3	<u>, </u>	^ ;	2	31(31)		70(71)	27	42	4	. 4
· ·	8	<u>c.</u>	0.7	S	26(25)	(67)// (5		χ̈́	7.7	: :	;
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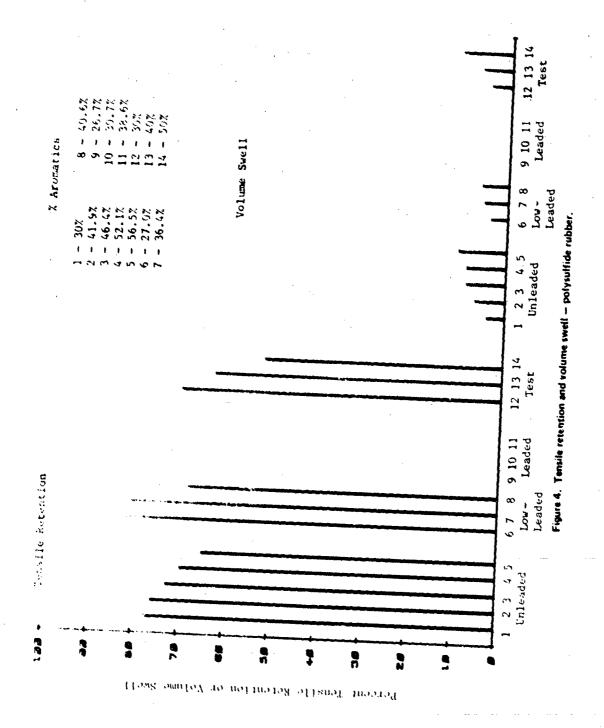
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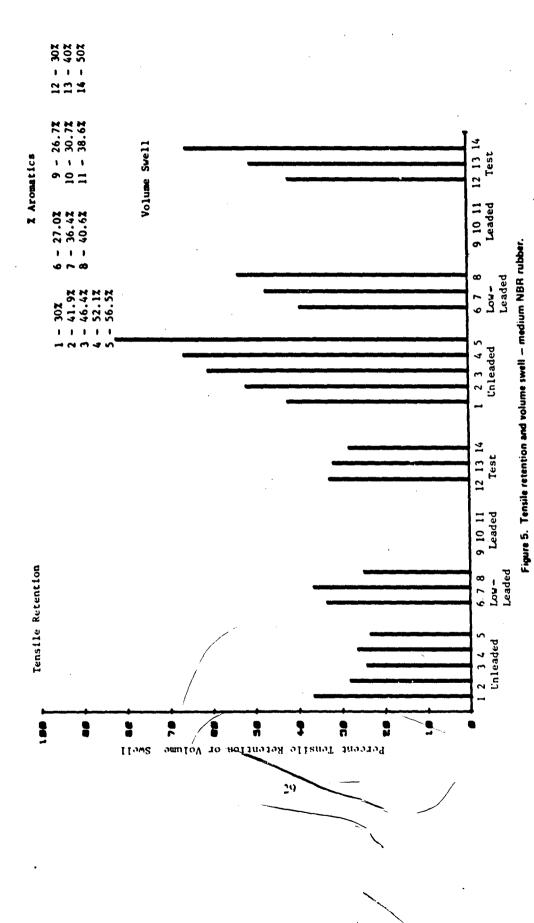
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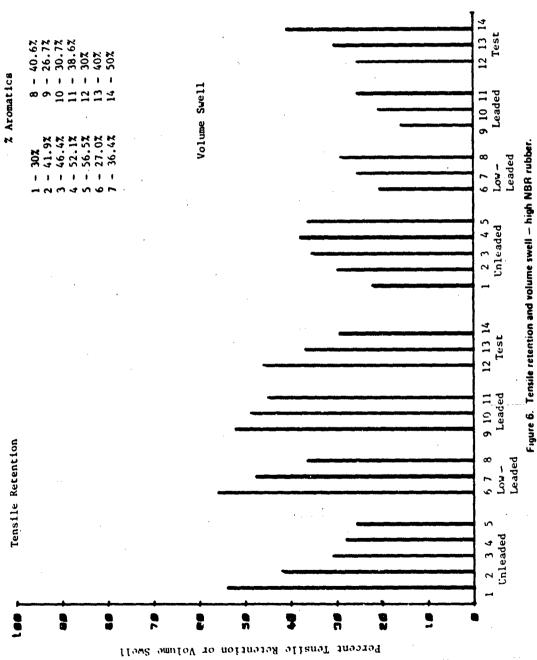












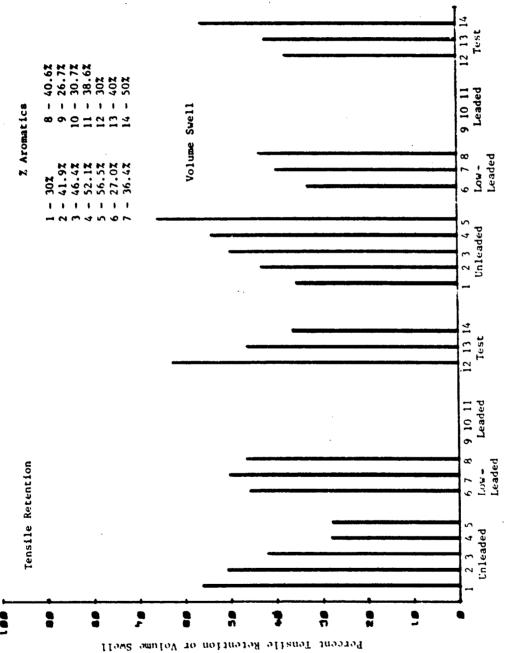


Figure 7. Tensile retention and volume swell - NBR/PVC rubber.

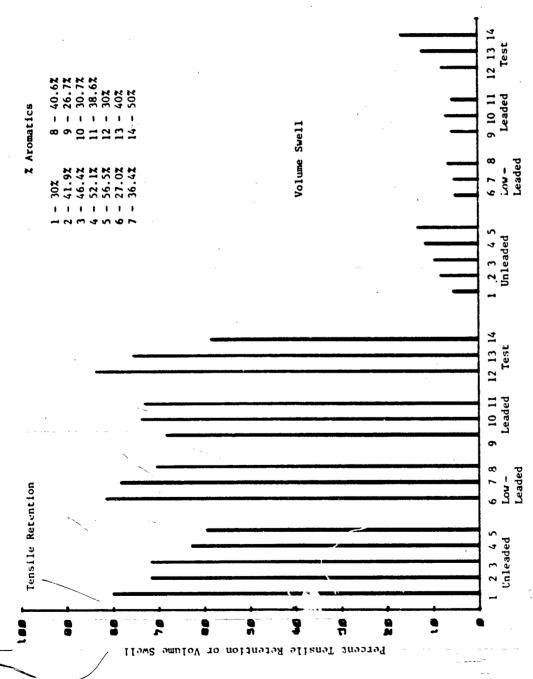
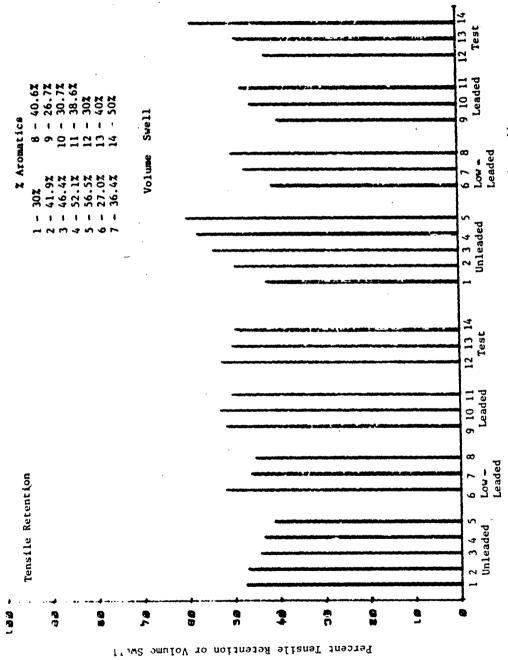


Figure 8. Tensile retention and volume swell - polyester urethane rubber.



Four of the five unleaded fuels, only one of the low-leaded fuels, and none of the leaded fuels compared contained an aromatic content of over 40%. When this factor is considered in evaluating the data in Figures 1 through 9 or in considering the whole spectrum of rubber/fuel combinations studied, the results fall in line with what one could predict. Higheraromatic content results in more severe swelling and deterioration, regardless of the presence or absence of lead. Likewise, the test fuels currently used remain fairly reliable indicators of performance. However, it becomes obvious that test fuels having 40%, 50%, or, perhaps, even 60% aromatic content must now be used to assure adequate evaluation of elastomer performance. Finally, among the representative elastomer types evaluated, ECO, polysulfide, high NBR, and the ester polyurethane are preferable choices if other requirements do not preclude their use.

Diffusion of the fuels through circular specimens of the nine rubbers generally follows the same pattern as the other properties evaluated. Higher aromatic content fuels escape through the rubber at a faster rate than those of low aromatic content. The low rate of fuel escape shown by the ester urethane is one significant reason why this material is now being used in fabrication of coated fabric fuel storage tanks in preference to the previously favored, but more porous, low and medium NBR's and neoprene.

7. Phase II. Extensive previous studies of the performance characteristics of fuel-resistant rubbers have shown that swelling and loss of tensile strength and elongation stabilizes after 48 to 96 hours' exposure. Prolonged exposure beyond 96 hours may produce further changes but they are of little significance. Seven days' exposure time was employed in Phase II tests to insure that all test fuel/rubber combinations had stabilized, particularly those having 60% and 70% aromatic content levels not generally employed previously. Figures 10, 11, and 12 compare the percentage loss of tensile strength and elongation and the increase in volume swell observed for all of the seven rubbers selected for this study. No diffusion testing was conducted in Phase II.

The elongation retention data of Figure 11 are a bit erratic and some anonalies are evident. However, the tensile retention data of Figure 10 and, particularly, the volume swell data of Figure 12 clearly show that there is no leveling-off or plateau effect. Degradation of strength and swelling continues at a rate proportionate to the total aromatic content of the test fuel. At the 60% aromatic content level (observed in some of the Phase I commercial fuels) volume swell of neoprene and low NBR, two of the most commonly used elastomer types, exceeds 100% and less than 20% of the original tensile strength is retained. Superiority of the polyester urethane is apparent and is further evidenced by the fact that even at the 100% aromatic level, the swell of this rubber is less than 60% (Table 15).

Percent Retention of Tensile Strength

12 345 Polyether Figure 10. Retention of tensile strength - 7-day immersion in test fuels. (Table 4) 12 345 High NBR £33393 30% Aromatics 40% Aromatics 50% Aromatics 60% Aromatics 70% Aromatics 70% Aromatics LOW NBR % Aromatics Neoprene 1 1 1 1 1 234 2 3 4 5 ECO 8 70 9 000 20

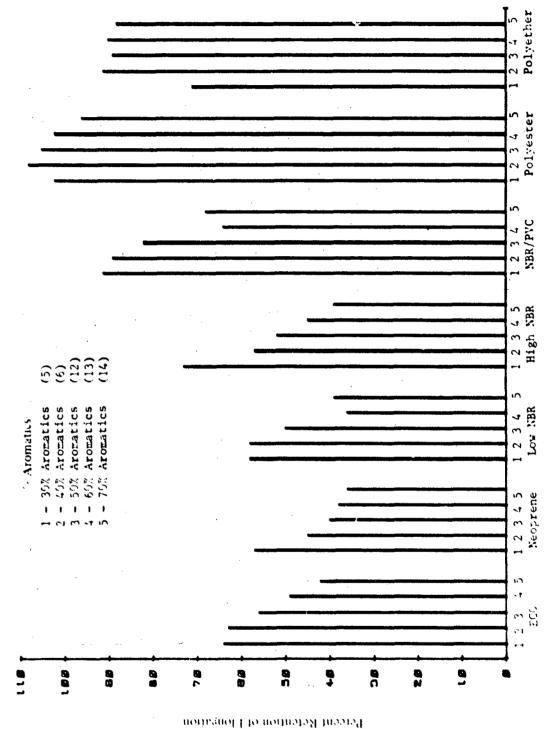
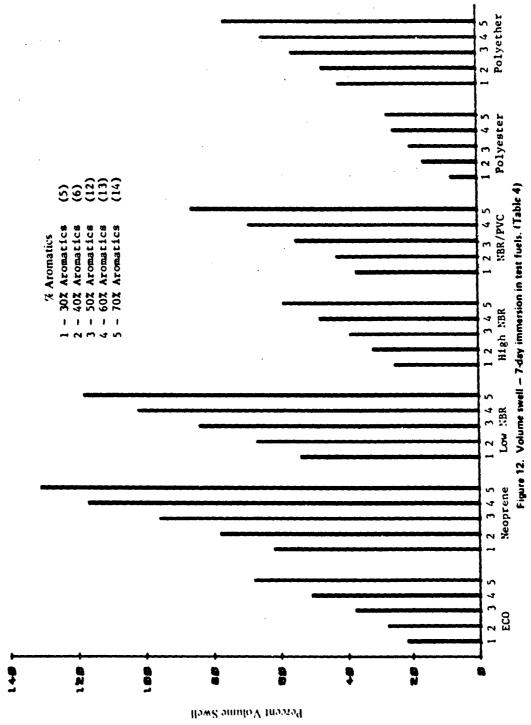


Figure 11. Retention of elongation - 7-day immersion in test fuels. (Table 4)



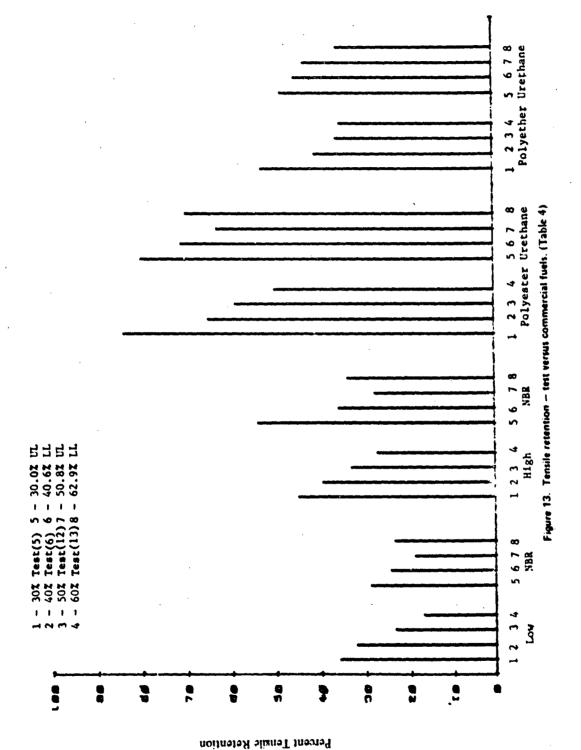


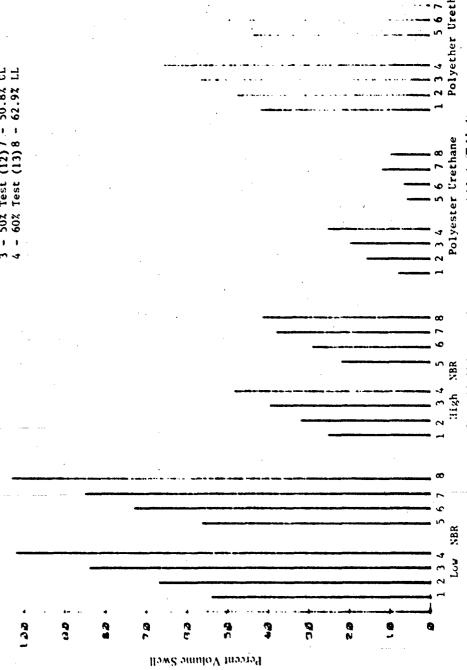
How well do these test fuels reflect performance characteristics of elastomeric compounds exposed directly in commercial fuels? This was examined by extracting data from the Phase I work which could be compared with results from Phase II. Data for the low and high NBR's and the two urethane rubbers exposed in unleaded fuels of 30% and 50.8% aromatic content and low-leaded fuels of 40.6% and 62.9% aromatic content were selected. Tensile strength retention and volume swell of these rubber/fuel combinations are shown in Figures 13 and 14, together with comparable data for the same rubbers exposed in the test fuels of 30%, 40%, 50%, and 60% aromatic content. Correlation of test and commercial fuel data is generally good, the only significant exception being the lower volume swell of the polyester urethane in the commercial fuels. This comparison again demonstrates that the presence or absence of lead in a commercial fuel, to which an elastomeric compound is exposed, is not important as the selection of test media having comparable or slightly higher aromatic content.

The issuance of directives forbidding the use of benzene in test fuels prompted further work to ascertain whether this carcinogen could be eliminated from Medium No. 5 of FTMS 601, Method 6001 and replaced with toluene or xylene. Volume swell testing was conducted, therefore, on six laboratory prepared test fuels having the following composition:

Isooctane	Toluene	Xylene	Benzene
(%)	(%)	(%)	(%)
60	40	0	0
60	25	15	0
60	20	15	5
60	15	20	5
60	20	5	15
60	5	15	20

Volume swell results for each of the seven Phase II rubbers exposed in the above test fuels are shown in Figure 15. No significant increase or decrease in volume swell attributable to variation in benzene content was observed. Any variance of less than 5% such as occurred here is easily within the span of allowable experimental error. These data also indicate that the necessity for inclusion of xylene in Medium No. 5 is questionable. A reference fuel consisting of 60% isooctane and 40% toluene would be adequate as a replacement for Medium No. 5 and as an additional 40% aromatic reference fuel in ASTM D-471.





qure 14. Volume swell — test versus commercial fuels. (Table 4)

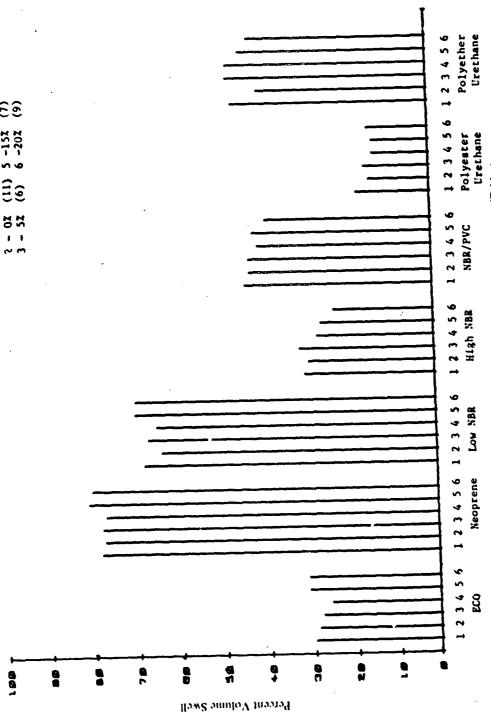


Figure 15. Volume swell versus benzene content - test fuels. (Table 4)

Data generated in the above benzene study and other Phase II work provided sufficient information to attempt prediction of the volume swell of randomly selected test fuels wherein the isooctane content was varied between 30% and 70% and the toluene, xylene, and benzene, between 0% and 50%. The composition of the constitutents of the tested fuels, expressed as a percentage of the total volume, together with observed volume swells for the seven Phase II rubbers formed a matrix of data input to a computer program for simplified regression analysis, written in the Basic language. Regression coefficients obtained were then used to calculate the predicted volume swell for untested rubber/fuel combinations. Results of this analysis are contained in Table 18. In order to spot check for accuracy, volume swells for nine selected untested rubber/fuel combinations were then determined, with the resultant values also included in Table 18.

Agreement between actual and predicted volume swell in the nine cases where the comparison was made was excellent. More important, however, is the fact that closer examination of the predicted volume swell data lends further support to the rationale for selecting a 60/40 isooctane/toluene mixture as a replacement for Medium No. 5. In all Phase I work, the benzene content of the commercial fuels never exceeded the 5% level of Medium No. 5. When the 40% aromatic content consisted entirely of benzene (No. 20 of Table 18), in three cases (ECO, neoprene, and low NBR), the predicted volume swell exceeded that of the test fuel containing 40% toluene (No. 8). However, when the test fuel contained only 40% xylene (No. 19), the predicted volume swell for ECO, low NBR, and the NBR/PVC rubbers was significantly lower and in none of the other cases did it exceed that of the 40% toluene test fuel by more than 4%. Finally, even when the prediction matrix was extended to include combinations of 60% aromatic content (Nos. 25 to 29), volume swell values were predominantly within the range of the ±5% allowable for experimental error.

IV. CONCLUSIONS

8. Conclusions. It is concluded that:

- a. The wide variations in type and level of aromatic constitutents of unleaded, low-leaded, and leaded commercial fuels preclude establishment of definite relationships between fuel composition and deleterious effects on rubber compounds beyond that which can be ascertained through use of laboratory reference or test fuels.
- b. The presence, absence, or concentration of lead in commercial fuels cannot be correlated with relative degradative effects on elastomeric compounds.

- c. Unleaded fuels by virtue of their generally higher aromatic content are somewhat more deleterious to rubber compounds, but anomalies, presumably related to the presence of other additives, may occur when comparisons are made with low-leaded or leaded fuels.
- d. Changes in physical properties of elastomeric compounds exposed in laboratory test fuels of increasing aromatic content (30-70%) occur at a rate proportionate to the aromatic content. No leveling-off or plateau effect is discernible.
- e. The benzene content of commercial fuels is less than 5%, a level low enough to justify elimination from laboratory test fuels in compliance with OSHA directives.
- f. Substitution of additional toluene for both xylene and benzene in Medium No. 5 of FTMS 601, Method 6001 will have no adverse effect on the reliability of estimating the deleterious effects of ruels on elastomeric compounds.
- g. A new reference fuel consisting of a 60/40 ratio of isooctane and toluene by volume should be included in both ASTM-D471 and FTMS 601, Method 6001 to fill the need for a 40% aromatic test medium.

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